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DESIGN OF AN IMPROVED COLUMN LEACHING APPARATUS FOR SEDIMENTS AND DREDGED MATERIAL

by

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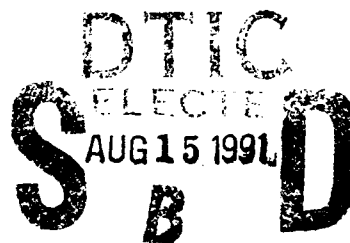Baton Rouge, Louisiana 70803

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This report describes a new column design for investigating contaminant leaching from sediments and dredged material. Improved sampling and sample preservation procedures are also described. A review of the literature on column leaching studies is also provided.

Three types of columns were reviewed: (a) soil columns, (b) solid waste columns, and (c) sediment and dredged material columns. The review focused on column design and operating parameters, not data interpretation. The literature review showed that the soil column literature is much more extensive than the column literature for either solid wastes or sediments and dredged material. A wide variety of column designs have been used, but nearly all involve a vertical column with a small diameter-to-length ratio, and percolation of water either upward or downward.

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    Information from selected studies on material used to fabricate columns,
column dimensions, hydraulic flux, direction of flow, type of soil, waste, or
sediment tested, and chemical(s) applied is tabulated and discussed, including
advantages and disadvantages of various column designs. The column literature on
sediments and dredged material indicated that the performance of column designs
based on the type of column used in most soil and solids waste studies is not
satisfactory for sediments and dredged material, primarily due to insufficient
flow.
    Based on the literature review and recommendations from a workshop held to
review sediment leaching studies, a thin-layer leaching column was designed. The
thin-layer column design has a large diameter-to-length ratio (25.4/4 cm) that
provides a short travel length and a large cross-sectional flow-through area.
The short travel length allows a greater number of pore volumes to be eluted
relative to previous designs, while the large cross-sectional flow-through area
provides sufficient volume of sample for chemical analysis. The improved design
includes upflow mode with distribution disks at both the top and bottom of the
column. Pore water velocity is controlled to be less than 1 E-05 cm/sec using a
constant volume pump.
    Improved sample collection and preservation are incorporated into the column
design. However, separate columns for investigating metals and organics are
required. For metals, an adaptation of the apparatus commonly used to study the
chemistry of soil and sediment suspension under controlled redox potential and pH
conditions is attached to the column outlet tube. This system collects and
preserves metals continuously by maintaining pH below 2.8 as the sample is
eluted. For organics, an adaptation of a Neilson-Kryger steam distillation
apparatus is attached to the column outlet tube. This system collects and
preserves organics continuously by use of a trapping solvent through which the
sample passes.

PREFACE

This report was prepared by Mr. Tommy E. Myers, Environmental Engineering Division (EED), Environmental Laboratory (EL), US Army Engineer Waterways Experiment Station (WES), Dr. Robert P. Gambrell, Laboratory for Wetland Soils and Sediments, Louisiana State University, and Dr. Marty E. Tittlebaum, Louisiana Water Resources Research Institute, Louisiana State University, for the Headquarters, US Army Corps of Engineers (HQUSACE). Drs. James M. Brannon, Ecosystem Research and Simulation Division, EL, and D. Dean Adrian, EED, were technical reviewers for this report. Funds for this work were provided by the Long-Term Effects of Dredging Operations (LEDO) Program. LEDO is managed as part of the Environmental Effects of Dredging Programs (EEDP) at WES. Dr. Robert M. Engler is Manager of EEDP. The HQUSACE Technical Monitors for LEDO were Mr. Dave Mathis, Mr. Joe Wilson, and Dr. William Klesch.

The work was conducted under the direct supervision of Mr. Norman R. Francingues, Jr., Chief, Water Supply and Waste Treatment Group, EED, and under the general supervision of Dr. Raymond L. Montgomery, Chief, EED, and Dr. John Harrison, Chief, EL.

Commander and Director of WES was COL Larry B. Fulton, EN. Dr. Robert W. Whalin was Technical Director.

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CONTENTS

DESIGN OF AN IMPROVED COLUMN LEACHING
APPARATUS FOR SEDIMENTS AND DREDGED MATERIAL

PART I: INTRODUCTION

Background

1. Column leach tests have become important tools in environmental engineering, particularly as laboratory models for simulating contaminant transport in porous media (Hamaker 1975; Helling and Dragun 1981; Rao and Jessup 1983; Brusseau and Rao 1989). Recently, column leach tests have been used by researchers at the US Army Engineer Waterways Experiment Station (WES) to provide information for evaluating the potential effects of leachate seepage when contaminated dredged material is disposed in confined disposal facilities (CDFs) (Environmental Laboratory 1987; Myers and Brannon 1988a; Palermo et al. 1989). These tests are still developmental and have not been adopted for routine application to sediments and dredged material.

2. A workshop organized to review the WES studies on sediment leaching recommended redesign of the column leach tests to include thin-layer columns and improved leachate collection systems (Myers and Brannon 1988b). The major problems with the current WES column test are the time required to conduct a column leach test and the potential for sample deterioration during leachate collection. Because of these concerns, an improved column leaching apparatus is needed.

Scope of Report

3. This report conceptually develops an improved column leaching apparatus for sediments and dredged material. Included is a review of the literature on column leach tests, a recommended column design, and recommended strategies for collecting and preserving column leachate.

PART II: LITERATURE REVIEW

Introduction

4.   Three areas of research involving column studies are reviewed in this report: soil columns, solid waste columns, and sediment and dredged material columns.   The soil column literature, which is much more extensive than that for either solid waste or sediments, is reviewed first.   Then the research involving solid waste columns is reviewed.   Solid wastes include municipal solid waste (domestic garbage) and industrial solid wastes, which may or may not be hazardous.   The sediment and dredged material column literature is rather limited and is reviewed last.

5.   This review focuses on column design and operating parameters such as flow rate and sample collection techniques.   Contaminant concentrations in leachate from column studies on soils, solid wastes, and sediments and dredged material are not discussed unless these data have direct bearing on developing an improved column design for sediments and dredged material.   Information from sediment studies was generally more useful.

Soil Column Studies

6.   The similarity of differential displacement of solutes in chromatographic columns to the movement of chemicals in soils has led to a wide range of laboratory column studies of solute transport in soils (Helling 1970; Hamaker 1975; Helling and Dragun 1981; Wagenet 1983; Rao and Jessup 1983; Brusseau and Rao 1989).   The soil column literature is extensive, and since much of the literature focuses on mathematical modeling, this review selectively considers representative work that describes typical soil column dimensions and operating conditions.

7.   Numerous soil column methods have been used, but nearly all involve a vertical column containing soil, a chemical introduced at the inlet, and percolation of water either upward or downward.   Information from selected studies on materials used to fabricate columns, column dimensions, pore water velocity, direction of flow, type of soil, and chemical applied is provided in Table 1.

Fabrication materials

8. Soil columns have been constructed with a variety of materials. Helling's review (1970) referred to aluminum rings, sectioned Monel metal pipe, stovepipe sections, rectangular plastic pipe, polyethylene bags, cellophane cylinders, glass, waxed paper columns, and paper carton cylinders. The more recent literature (Table 1) indicates that synthetics continue to be a popular choice for fabricating soil columns. Use of glass and steel is less common. Unconventional fabrication techniques were reported by Goerlitz (1984) and Wierenga et al. (1975). Goerlitz (1984) prepared soil columns using stainless steel chromatography columns so that the eluent could be fed directly to the detectors of a liquid chromatograph. Wierenga et al. (1975) used Techite, a composite of fiberglass, polyester resin, and sand to fabricate large soil columns (75 cm inside diameter (ID) by 150 cm).

9. The basis for selecting a particular material from which to fabricate a soil column is not discussed in the literature. It seems to be a matter of preference and convenience. Apparently, the solute being studied has little bearing on the selection of materials from which to fabricate soil columns.

Column dimensions

10. A diameter-to-length ratio of less than 1 is a geometric feature of cylinders that most of the soil column studies in Table 1 use. Hoffman and Rolston (1980) and Nkedi-Kizza et al. (1983), however, used columns with diameter-to-length ratios that were greater than 1. Column diameters range from the small diameter (1.5 cm ID) stainless steel columns used by Goerlitz (1984) to the large diameter (75 cm ID) columns used by Wierenga et al. (1975) and O'Connor et al. (1980). The column length listed most often in Table 1 is 30 cm. This is the length recommended in US Environmental Protection Agency guidelines for pesticide mobility testing (US Environmental Protection Agency 1978). The guidelines do not include a specification for column diameter.

11. Some investigators have conducted studies using more than one column length. Wagenet, Biggar, and Nielsen (1977) used 7.6-cm ID columns of 15- and 30-cm lengths to investigate transport and biological transformation of urea fertilizer in soil. The authors noted no differences in transport and transformation processes between the two columns. Hoffman and Rolston (1980) investigated transport of organic phosphate in soil using 7.5-cm ID columns 2.4 to 5.1 cm in length. They noted some experimental problems when pulsing the short columns.

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12. Most of the studies listed in Table 1 involve small diameter col-
umns that are less than 10 cm in diameter. The exceptions are the studies by
Cassel et al. (1974), Wierenga et al. (1975), and O'Connor et al. (1980). The
large soil columns have been used to simulate the complexities of field trans-
port processes. Cassel et al. (1974) investigated the displacement of nitrate
and chloride in large (30 cm ID by 80 cm) soil cores (disturbed and undis-
turbed) in order to obtain information on behavior of solutes under field
conditions. Wierenga et al. (1975) compared predicted salt distributions and
those observed in large columns (75 cm ID by 150 cm) in which field irrigation
was simulated. O'Connor et al. (1980) conducted a similar study of the move-
ment of 2,4,5-trichlorophenoxyacetic acid under conditions representing field
irrigation.

13. The distinction between small columns and large columns is not only
one of scale but also of complexity and control. In small laboratory columns,
transport processes can be isolated from the variability that characterizes
the environment so that basic mechanisms can be studied under carefully con-
trolled conditions. In large columns, environmental complexities that are
best studied at a large scale can be simulated.

14. No studies of the effect(s) of column dimensions on contaminant
elution characteristics were found during this literature review. Although
there is a potential for wall effects in rigid-wall apparatus (McNeal and
Reeve 1964; Dudgeon 1967; McIntyre et al. 1979; Sommerton and Wood 1988), the
soil column literature in Table 1 usually makes no mention of wall effects.
From the standpoint of minimizing wall effects, columns should have large
diameter-to-length ratios (Helling and Dragun 1981), or the column-diameter-
to-particle-diameter ratio should be on the order of 200:1 or greater
(Sommerton and Wood 1988).

Flow

15. Because unsaturated flow is difficult to control and model, most of
the laboratory column studies listed in Table 1 involve saturated flow. Down-
flow was used more often than upflow. Flow is controlled with a constant
volume pump or a constant head device. Pore water velocities were
1 E-05 cm/sec, or greater.

16. Pore water velocity not only affects chemical elution in porous
media by bulk movement of water but also by affecting the nature of chemical
interactions between solid and aqueous phases (Rubin 1983; Valocchi 1985; Bahr
and Rubin 1987; Brusseau and Rao 1989). If the rates of reaction between

solid and aqueous phases are fast relative to the bulk movement of water, a
local chemical equilibrium between water and solids is established. Non-
equilibrium processes govern when the convective transport of water is fast
relative to the rates of reaction. Pore water velocity is therefore a key
parameter in soil column studies.

Solid Waste Column Studies

17. Solid waste column studies have been conducted to provide data for
estimating the characteristics of leachate at disposal sites and/or measure
the amount of contaminant that can be leached from a waste. Numerous column
methods have been used to investigate solid waste leaching, but nearly all
involve percolating clean water through a vertical column of waste. Informa-
tion from selected studies on materials used to fabricate columns, column
dimensions, water application rates, direction of flow, and waste type is
provided in Table 2.

Fabrication materials

18. Columns for leaching solid wastes have been constructed with a
variety of materials including glass, plastic, steel, Plexiglas, and concrete
(Table 2). Selection of materials from which to fabricate a solid waste
leaching column has been based on the size of the column. Glass and plastic
have been used to fabricate small laboratory-type columns, and steel and
concrete have been used to fabricate large pilot-scale leaching columns.
Special finishes have been applied to the interior column walls of pilot-scale
leaching columns containing municipal solid waste (MSW) in order to protect
the walls from the corrosive action of MSW leachate (Steng 1977; Fungaroli and
Steiner 1979; Myers et al. 1979; Walsh and Kinman 1979; Francis, Maskarinec,
and Goyert 1986).

Column dimensions

19. The dimensions of a solid waste leaching column depend on the par-
ticle size characteristics of the waste. Co-disposal studies involving MSW
have been conducted in large, pilot-scale columns (Steng 1977; Fungaroli and
Steiner 1979; Myers et al. 1979; Walsh and Kinman 1979; Francis, Maskarinec,
and Goyert 1986; Pohland 1987) because MSW contains objects too large for
placement in small laboratory columns. Solid waste leaching in small-diameter
columns is feasible when the waste particle size is relatively small, such as
is the case with fly ash (Dodd et al. 1981; Jackson, Benedik, and Jackson

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1981; Young et al. 1984). Stevens, Jenkins, and Wilson (1983) used 15-cm ID columns to study the attenuation of pesticides and herbicides by pulverized MSW.

Flow

20. Table 2 lists water application rates as the average hydraulic flux (if water were continuously applied) instead of pore water velocity. Pore water velocity is not a meaningful parameter for heterogeneous media of unde-termined porosity and degree of saturation such as MSW.

21. Water application rates have varied widely depending on the objec-tives of the individual investigator. Steng (1977), Fungaroli and Steiner (1979), Myers et al. (1979), Walsh and Kinman (1979), Dodd et al. (1981), Stevens, Jenkins, and Wilson (1983), and Pohland (1987) used rates of typical annual rainfall for the contiguous United States. Other investigators (Darcel 1983; Jackson, Garrett, and Bishop 1984; and Young et al. 1984) applied water at much higher rates. Equilibrium versus non-equilibrium leaching issues (as dealt with extensively in the soil column literature) have not been a consid-eration in most solid waste column leaching studies.

22. Interrupted flow in which water is applied periodically, typically once a week, was a common practice for pilot-scale columns containing MSW. In most cases, materials to be leached were loaded into columns in an unsaturated condition. One aspect of using initially dry or partially saturated material is that leachate will not flow from the column until the lowest portion is sufficiently wet. Pohland (1987) added water to bring MSW immediately to field capacity and then added water weekly to simulate leachate production from MSW. Fungaroli and Steiner (1979) and Walsh and Kinman (1979) applied water at variable rates to simulate monthly variations in infiltration rates for selected locales in the United States. Walsh and Kinman (1979) also exam-ined extremes in average annual infiltration rates.

Sediment and Dredged Material Column Studies

23. There have been relatively few studies of sediment or dredged mate-rial leaching in laboratory columns. In general, little is known about the formation of leachate when contaminated dredged material is disposed in CDFs. Some work was accomplished under the Dredged Material Research Program (Mang et al. 1978), and more recently laboratory column studies of sediment leaching

have been conducted at WES (Myers and Brannon 1988b). The available information is reviewed below.

Mang et al. 1978

24. Mang et al. (1978) investigated the generation of leachate from 16 large Plexiglas columns under various environmental conditions. The study used dredged material from CDFs located near Mobile, AL, Sayreville, NJ, Grand Haven, MI, Seattle, WA, and Houston, TX. The columns were 29 cm ID, and dredged material was placed to a depth of 30 cm (Figure 1). Soils were placed beneath the dredged material in order to investigate contaminant attenuation by foundation soils at CDFs.

25. Measured amounts of influent were added at the top of the columns which were open to the air and allowed to flow by gravity to leachate collection bottles. The collection bottles were kept anaerobic by a constant nitrogen gas purge and water lock seal. Specific flow rates varied from 0.026 to 0.10 cm/d, depending on the hydraulic conductivity of the dredged material.

26. Various leaching solutions were used including distilled water, distilled water acidified to pH 4.5 with sulfur dioxide, hard water buffered with bicarbonate, and leachate obtained from a solid waste landfill. Leachates were analyzed for major cations and anions, heavy metals, polychlorinated biphenyls (PCBs), chlorinated pesticides, nutrients, pH, Eh, alkalinity, and conductivity.

27. Due to the low hydraulic conductivities of the dredged materials, less leachate than was needed for complete chemical analysis (2 ℓ) was sometimes collected. In the few instances where flow exceeded the target flow, flow control was accomplished by raising the leachate collection bottle (Figure 1). Moisture content in the columns was variable in this study. In most of the columns, there were regions of unsaturated and saturated flow. Pore water samples were also collected from about mid-depth of the dredged material by applying vacuum to interstitial sampling ports (Figure 1). The desired volume of pore water (500 ml) was not always collected.

28. Collection of sufficient sample for chemical analysis was a major problem in this study. According to the authors, it was very difficult to obtain conclusive results from the column experiments because sufficient sample for chemical analysis could not be obtained on a consistent basis.

29. The authors also noted variation in oxygen penetration among the dredged materials studied. Eh data showed that in some columns oxygen penetrated throughout the dredged material and in others there was very little

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PLEXIGLAS LYSIMETER COLUMN

ADJUSTABLE HEIGHT SAMPLE COLLECTION RACK TO CONTROL RATE OF FLOW

LEACHING FLUID

DREDGED MATERIAL

INTERSTITIAL PORE SAMPLER

N$_2$ GAS

INTERFACING SOIL

POLYPROPYLENE BEADS SUPPORT BENCH
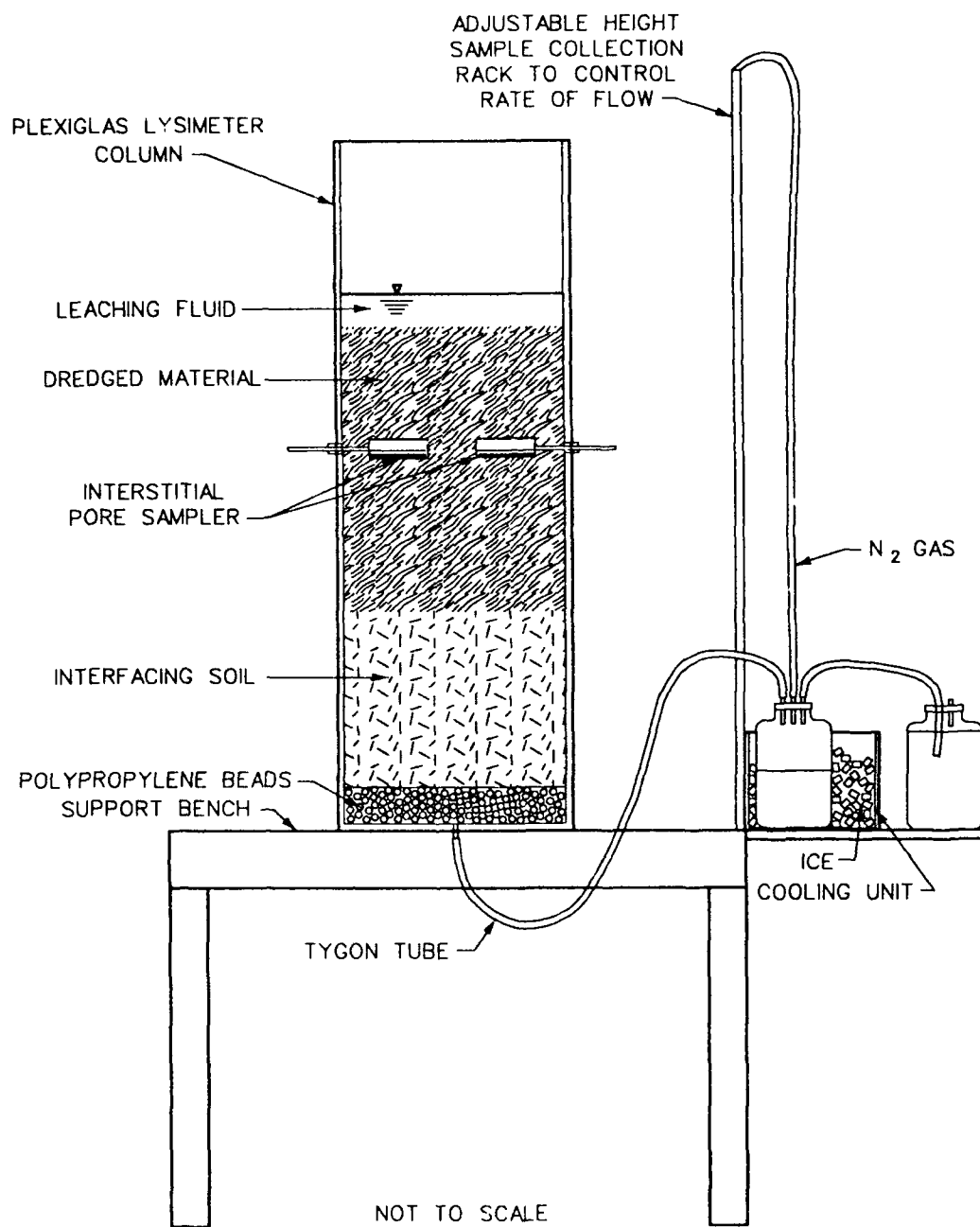
ICE

COOLING UNIT

TYGON TUBE

NOT TO SCALE

Figure 1. Lysimeter column experimental setup
(from Mang et al. 1978)

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penetration. Differences in oxygen penetration were related to differences in organic carbon content of the dredged material and degree of saturation.

WES laboratory column studies

30. The WES conducted batch and column leaching studies on contaminated sediment from Indiana Harbor, Indiana (Environmental Laboratory 1987), Everett Harbor, Washington (Palermo et al. 1989), and New Bedford Harbor, Massachusetts (Myers and Brannon 1988a). These studies attempted to couple chemical processes governing interphase transfer of contaminants from the sediment solids to pore water with the fluid mechanics of porous media flow. The test procedures and experimental design were based on mass transport theory as outlined in Hill, Myers, and Brannon (1988).

31. Each study involved continuous-flow leaching (saturated conditions) in divided-flow, stainless steel columns designed to minimize wall effects (Figures 2 and 3). The double-ring design in the baseplate (Figure 3) divides flow, separating the water flowing through the center of the column from that flowing down the walls. Column ID was 15.2 cm, and the diameter of the flow-through area for leachate collection and analysis was 9.8 cm. Separate columns in triplicate were set up for obtaining leachate for metal and organic contaminant analysis. Separate columns were used for leaching metals and organics because of differences in the volume of leachate required for metals (0.1 ℓ) and organic analyses (1 ℓ). Sediment height was 18 cm in columns used to obtain leachate for metal analysis and 36 cm in columns used to obtain leachate for organic analysis.

32. Sediment was added to the columns in 5-cm lifts in the as-received condition, that is, water-saturated mud. As each lift was added, the column was vigorously agitated on a vibrating table. Once the columns were in operation, consolidation reduced porosity and increased resistance to flow. Flow was regulated by adjusting the pressure in the head space gas during testing. Average pore water velocities were in the range of 1 E-06 to 1 E-05 cm/sec. Initial operating pressures were 0.0 psig, and depending on the sediment column height (type of contaminant), final operating pressures ranged from 15 to 30 psig (103 to 207 kPa gage). Hydraulic conductivities ranged from 1 E-08 to 1 E-06 cm/sec.

33. Sediments were leached with distilled deionized water. Leachate from the center tap on the column was collected in graduated 1,000-ml glass cylinders. The collection vessels were isolated from the atmosphere by a constant nitrogen gas purge and a water trap (Figure 4). Leachate from

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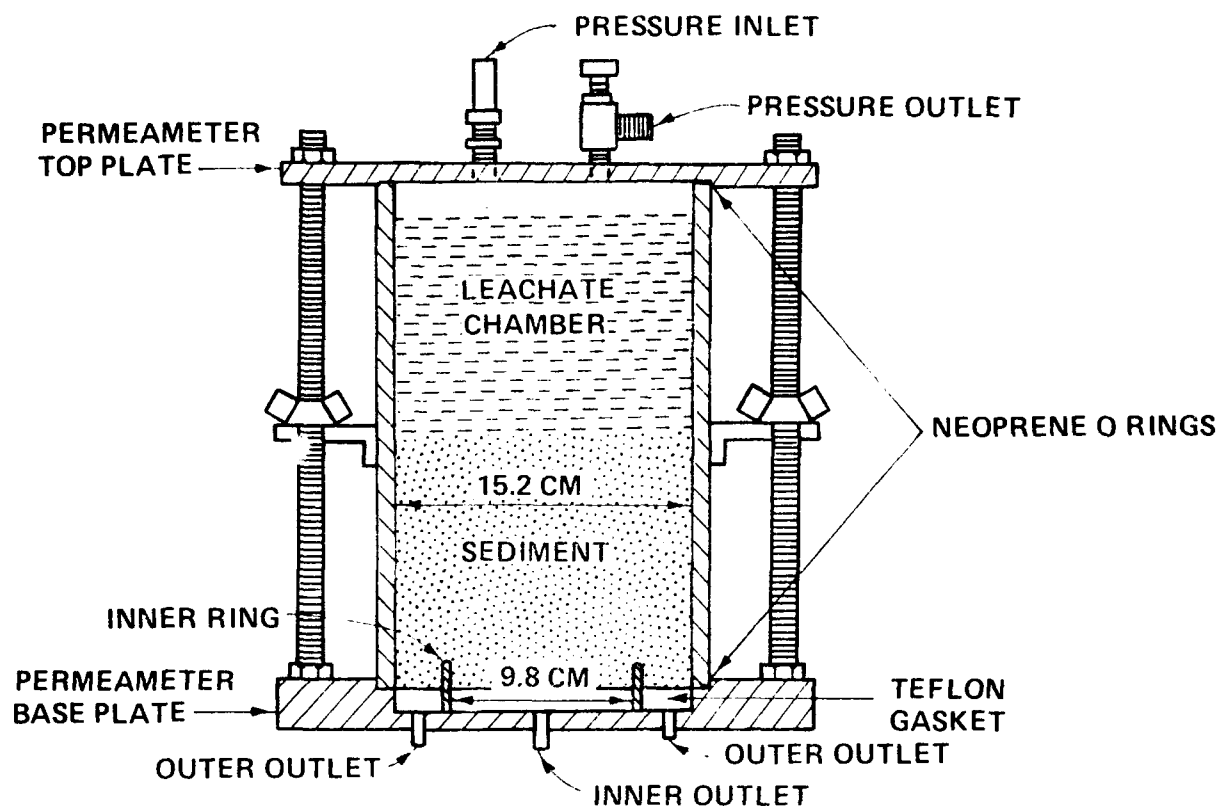
Figure 2.   Divided-flow permeameter for sediments
(from Myers and Brannon 1988b)
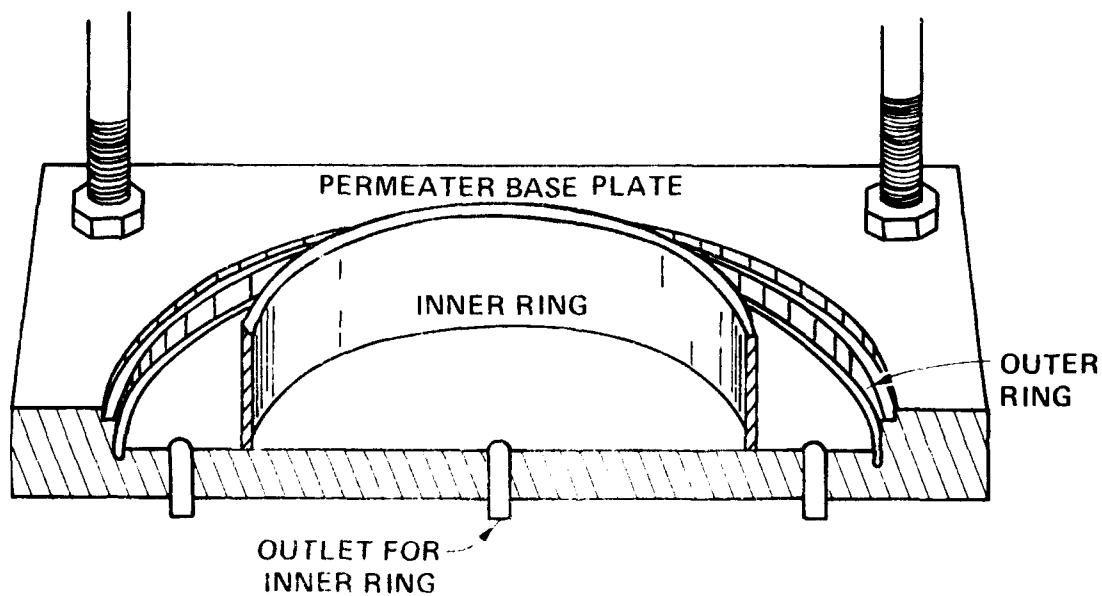
Figure 3.   Schematic of divided-flow permeameter baseplate

LEACHATE

VENT TO
ATMOSPHERE

REGULATOR

GAGE

TEFLON
TUBING

COPPER
GAS LINE

TEFLON
CORK

NOT TO SCALE

WATER TRAP   PERMEAMETERS   BOTTLED
                                          NITROGEN
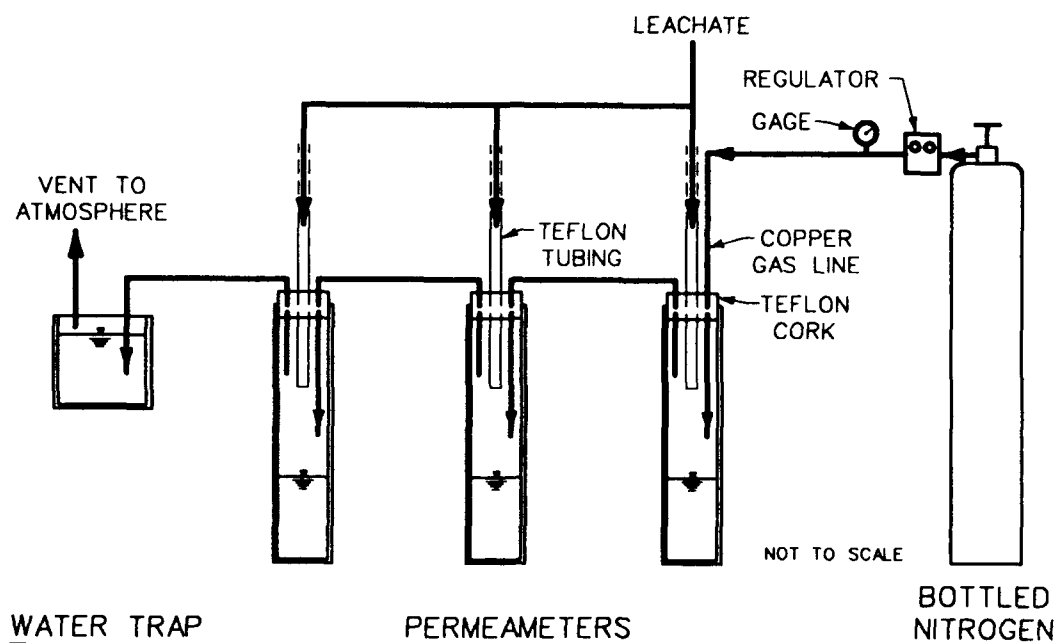
Figure 4.   Isolation system for leachate from divided-
flow permeameters (from Myers and Brannon 1988a)

columns set up for metal leaching was analyzed for pH, conductivity, dissolved
organic carbon, and selected metals. Leachate from columns set up for inves-
tigating leaching of organics was analyzed for pH, conductivity, dissolved
organic carbon, polychlorinated biphenyls, and other organic chemicals. Sam-
ples were filtered under nitrogen through 1.0-μm nominal pore size glass fiber
filters prior to analysis.

34.   Column leaching studies were also conducted on sediment that had
been aged by exposure to air at ambient temperatures for 6 months (Environ-
mental Laboratory 1987; Palermo et al. 1989; Myers and Brannon 1988a). The
experimental conditions were the same as for untreated sediment except that
air was used as the head space gas, and samples were filtered in an air
environment.

Advantages and disad-
vantages of current designs

35.   Mang et al. (1978). The column used by Mang et al. (1978) includes
attenuation by foundation soils and flow is by gravity. The major advantage
of the Mang et al. (1978) design is that it models some of the important envi-
ronmental factors affecting contaminant migration from CDFs by leachate
seepage.

36.   The major disadvantage of the design used by Mang et al. (1978) is
the realistic flow conditions. Because the column simulates leaching on a

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time scale that approaches the natural setting, the column is of little practical use for investigating long-term trends. Another disadvantage of the flow regime is that it is very difficult to use theoretical analysis of column elution trends to investigate fundamental processes when saturated and unsaturated flow are present. In addition, the oxygen penetration associated with unsaturated flow indicates that the column probably simulates leaching in the upper, unsaturated zone in a CDF. It is questionable if the columns used by Mang et al. (1978) simulate leaching in the saturated zone of a CDF, the zone of primary interest when contaminant transport by leachate seepage is a concern.

37. <u>Myers and Brannon (1988b).</u> The WES sediment column (Myers and Brannon 1988b) was designed for the investigation of basic processes (bulk flow, hydrodynamic dispersion, and desorption) so that mass transport theory can be applied and used to investigate fundamental processes and predict long-term leaching trends. The major advantage of the WES column is that it provides accelerated leaching data for verification of desorption coefficients determined in batch leaching tests.

38. The Myers and Brannon (1988b) column, however, does not fully implement the design objectives. As indicated by the pressure variations during testing (Figure 5), constant flow was difficult to maintain. In addition, due to the low hydraulic conductivities encountered, it has been difficult to elute the number of column pore volumes that batch leach tests with saltwater sediments indicate are needed to fully investigate long-term trends in contaminant release.

39. To increase the number of pore volumes eluted in a given period of time, the column length must be reduced or the pore water velocity must be increased, or both. However, as discussed in the review of soil column studies, pore water velocity affects the processes controlling contaminant release in porous media (Rubin 1983; Valocchi 1985; Bahr and Rubin 1987; Brusseau and Rao 1989). Adjustments of pore water velocity, therefore, should be made cautiously and judiciously. With the current design, column lengths range from 14 to 35 cm depending on the type of contaminant, and pore water velocities are in the range of 1 E-06 to 1 E-05 cm/sec. For these conditions, a year or more is required to conduct a column leach test. This amount of time limits the usefulness of the test and increases costs.
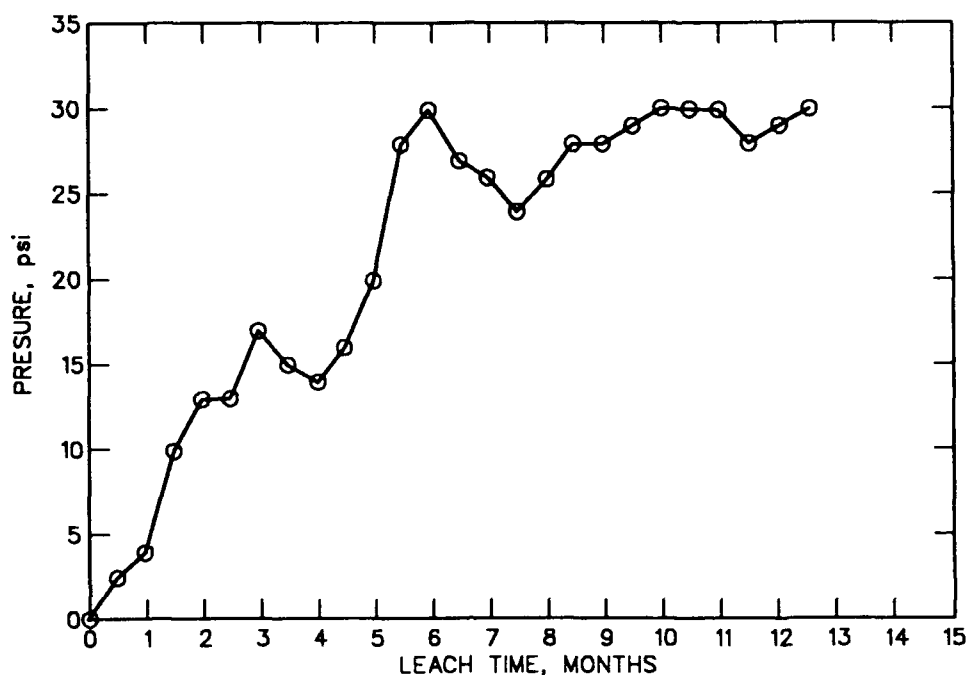
Figure 5. Pressures required to maintain constant flow in
divided-flow permeameters for anaerobic leaching of organics
(to convert pounds (force) per square inch to kilopascals,
multiply by 6.895 (Myers and Brannon 1988a))

40. Because of the slow percolation rates, the Myers and Brannon
(1988b) apparatus collects and holds leachate samples for periods of 2 weeks
or more. During this holding period, the integrity of the sample can be com-
promised by sorption to the walls of the collection vessel and volatilization
into the vessel head space. The slow purge with nitrogen gas would continu-
ously remove contaminants in the vessel head space. For these reasons, there
has been concern about the integrity of the column leachates that were col-
lected and analyzed in previous studies.

PART III: IMPROVED COLUMN DESIGN

Introduction

41. As previously discussed, the major problems with the Myers and Brannon (1988b) column leaching apparatus are the time required to conduct a column leaching study and the potential for sample deterioration during leachate collection. The first problem is addressed in this part of the report.

42. A workshop organized to review the WES sediment leaching studies recommended leaching a sediment sample whose thickness is small relative to its diameter (Myers and Brannon 1988b). By reducing the distance water has to travel, more pore volumes can be eluted in a given period of time for the same pore water velocities used in previous studies. No other major recommendations for redesign and operation of a column leaching apparatus for sediments were provided except for the recommendations regarding sample collection and preservation.

43. Certain features of the previous design were therefore retained in the improved design described in this part of the report. The key features retained from the previous design are as follows:

   a. Column leach tests are conducted to simulate leaching anaerobic sediment using saturated flow.

   b. Leaching columns are fabricated using stainless steel.

   c. Flow is controlled such that average pore water velocities are in the range previously used or lower.

   d. Columns are designed to minimize wall effects.

   e. Separate columns are operated for leaching of metals and organics.

Design Constraints

44. Based on theoretical considerations and the experience accumulated in previous studies, the following constraints on the design process were established:

   a. Average pore water velocity should not exceed 1 E-05 cm/sec.

   b. Ten pore volumes of water should be eluted in 6 months or less.

   c. Flow should be sufficient to produce samples of sufficient size for chemical analysis. Sample size should be 1 ℓ or more.

45. The first constraint is needed because pore water velocity affects the chemical nature of the desorption reaction(s). Desorption can be either equilibrium or kinetically controlled, depending on a complex interplay between fluid mechanics and desorption chemistry (Rubin 1983; Valocchi 1985; Bahr and Rubin 1987). At anticipated pore water velocities for dredged material in a CDF, chemical kinetics should approach equilibrium. Average pore water velocity in a column leach test for sediment or dredged material should not, therefore, exceed the limit for which desorption is equilibrium controlled. Within this constraint, column pore water velocities can be accelerated relative to pore water velocities in a CDF without changing the nature of desorption and, hence, the characteristics of contaminant elution curves. The upper limit on pore water velocity is not known or cannot be calculated a priori because desorption kinetics are not well understood. Some laboratory soil column studies have shown equilibrium-controlled desorption to be a valid assumption at pore water velocities as high as 1 E-04 cm/sec (Valocchi 1985). Most soil column studies, however, indicate non-equilibrium effects at pore water velocities greater than 1 E-05 cm/sec. It is therefore recommended that the average pore water velocity in column leach tests for sediments and dredged material be less than 1 E-05 cm/sec.

46. The second constraint is based on batch desorption trends observed for saltwater sediments (Palermo et al. 1989; Myers and Brannon 1988a). These studies indicated that elution of 10 pore volumes is needed to verify trends observed in batch leach tests. A 6-month time frame for eluting 10 pore volumes was arbitrarily set. Three to five months are usually required for complete chemical analysis of leachate samples and several months may be required for data reduction and report preparation. Completion of column leaching in 6 months or less is a target anticipated to be consistent with the pre-project data needs of most dredging projects involving contaminated sediments.

47. The third constraint is based on sample requirements for analysis at WES. At least 1 ℓ of sample is required for organic analysis. Metals analysis requires significantly less sample. Although separate columns will be operated for leaching of metals and organics, separate designs are not necessary since a column design that meets the constraints for organics will also be satisfactory for leaching metals.

Design Calculations

48. The basic equation for flow through porous media is the Darcy equation (McWhorter and Sunada 1977) which is as follows:

$$q = - K \, (dh/dl) \tag{1}$$

where

$\qquad$ q = Darcy velocity, cm/sec

$\qquad$ K = hydraulic conductivity, cm/sec

$\qquad$ (dh/dl) = hydraulic gradient, dimensionless

Darcy velocity is the flow per unit of bulk area. Bulk area includes both pore space and solids. Other names are commonly given to the Darcy velocity such as approach velocity, hydraulic flux, Darcy flux, and surficial velocity. The average pore water velocity is another velocity given by

$$v = q/n \tag{2}$$

where

$\qquad$ v = average pore water velocity, cm/sec

$\qquad$ n = porosity, dimensionless

Flow in a column is given by

$$Q = A \, q = A \, v \, n$$

$$\tag{3}$$

$$Q = (\pi \, D^2/4) \, v \, n$$

where

$\qquad$ Q = flow, cm^3/sec

$\qquad$ A = cross-sectional area of the column, cm^2

$\qquad$ π = 3.14159...

$\qquad$ D = column diameter, cm

The number of pore volumes eluted for a time period t is given by

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$$P_v = v\,t/Z$$

so that

$$v = P_v\,Z/t \qquad\qquad (4)$$

where

P_v = number of pore volumes eluted

Z = length of the column, cm

Combining Equations 3 and 4 yields

$$Q = (\pi\,D^2\,P_v\,Z\,n)/(4\,t) \qquad\qquad (5a)$$

and after rearranging

$$Z\,D^2 = (4\,Q\,t)/(\pi\,n\,P_v) \qquad\qquad (5b)$$

Given values for Q, t, n, and P_v, there is an infinite set of $[Z,D]$ pairs that satisfy Equation 5b.

49. In leaching studies on contaminated sediments (Environmental Laboratory 1987; Myers and Brannon 1988a; Palermo et al. 1989), porosity ranged from 0.6 to 0.8. For design purposes, a porosity of 0.7 is used. Assuming a 2-week sampling period, the flow Q should be 1,000 ml per 2 weeks (13 ℓ per 6 months) to satisfy the constraint on sample size. The time period t is therefore 6 months, and the number of pore volumes eluted is 10 (0.769 pore volumes every 2 weeks). Table 3 lists selected $[Z,D]$ pairs calculated using the values for Q, t, n, and P_v given above.

50. Design constraints (b) and (c) are satisfied by the $[Z,D]$ pairs listed in Table 3. From this list, those pairs which satisfy the constraint on average pore water velocity were determined using Equation 4. The average pore water velocities listed in Table 3 show that column lengths of 12 cm or less satisfy the constraint on average pore water velocity.

51. If more frequent sampling is needed, then the diameter of the column will have to be increased in order to satisfy all constraints. Consider, for example, a sampling program that calls for collecting 1,000 ml of leachate

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each week. The flow in this case is 26 ℓ per 6 months. The other parameters (t, P_v, and n) remain the same. Table 4 shows the column dimensions required for elution of 1,000 ml of leachate per week and 10 pore volumes per 6 months. Since each weekly sample of 1,000 ml represents 0.385 pore volumes, the number of data points on an elution curve prepared with this sampling scheme will be twice the number available with the biweekly sampling scheme involving a smaller diameter column. Note that the average pore water velocity is unchanged since the number of pore volumes eluted is the same for both cases.

52. The calculations in Tables 3 and 4 show that, depending on column length, there are several choices for column diameter that are acceptable. A column diameter of 20.3, 25.4, or 30.5 cm (8, 10, or 12-in.) is recommended because these are standard pipe sizes that should be available as stock items. For $Q = 13$ $\ell/6$ months, $t = 6$ months, $n = 0.7$, and $P_v = 10$, Equation 5b yields $Z = 5.7$, 3.7, and 2.5 cm for $D = 20.3$, 25.4, and 30.5 cm, respectively. Theoretically, any one of these Z,D pairs should be satisfactory.

53. However, in selecting a column length, consideration must also be given to potential problems with channelization. As column length decreases, problems with early breakthrough due to preferential flow through a few large channels increases. Calculations for column lengths less than 4 cm were not provided in Tables 3 and 4 because of concern about channelization. A minimum column length of 4 cm is recommended as a good tradeoff between potential for channelization problems and minimization of average pore water velocity. From the previous consideration of standard pipe diameters and theoretical column lengths, the standard pipe size with a theoretical column length closest to 4 cm is $D = 25.4$ cm. It is therefore recommended that a column 25.4 cm in diameter and 4 cm in length be used as a prototype for testing and development of a final column design.

54. Since the recommended Z has been increased over the value initially provided by Equation 5b for $D = 2.54$ cm, the flow required to elute 10 pore volumes in 6 months must be recalculated using Equation 5a. For $Z = 4$ cm, $D = 25.4$ cm, $P_v = 10$, $n = 0.7$, and $t = 6$ months, Equation 5a yields $Q = 14,187$ cm^3/6 months. From Equation 3, the average pore water velocity is 2.6 E-6 cm/sec. This pore water velocity is within the design constraints. For $Z = 4$ cm, $D = 25.4$ cm, and $n = 0.7$, the void volume of the column is 1,419 cm^3. Every 14 days, 1,115 cm^3 of leachate will be eluted, representing 0.786 pore volumes. This 2-week flow will provide ample volume of sample for chemical analysis.

Other Design Considerations

Flow

55. It is recommended that an upflow mode using a constant-volume pump be used. This mode of operation will provide better flow control than that possible using downflow with variable head. In the Myers and Brannon (1988b) column, flow was controlled by manual adjustment of operating pressure. Because pressure adjustment could not be made continuously, there were times when flow was much lower and times when flow was much higher than the average flow calculated for an entire study period. Upflow accomplished using a constant-volume pump will tend to minimize consolidation effects and eliminate variations in flow. This is an important advantage because the theory used to model column elution assumes flow is constant and neglects consolidation. Thus, with upflow and a constant-volume pump, experimental conditions will more closely approximate model assumptions.

56. The operating head that the pump will have to provide depends on the hydraulic conductivity of the sediment and the length of the column. Previous work with contaminated sediments has shown that sediment hydraulic conductivities in the range of 1 E-08 to 1 E-06 cm/sec can be expected. From Equation 1, the range in operating head (referenced to atmospheric pressure) for a pump providing a Darcy velocity of 1.8 E-06 cm/sec through a 4-cm column containing sediment with hydraulic conductivities in the range of 1 E-08 to 1 E-06 cm/sec is 7.2 to 720 cm of water (0.1 to 10.2 psi$g$), respectively. The pump will have to provide a flow of 0.08 cm^3/min over this range in operating head.

Inlet-outlet conditions

57. It is recommended that distribution disks be used to distribute flow at the bottom and tops of the sediment column. The disks should be sintered stainless steel or glass or some other inert material. The end plates in which the distribution disks are embedded should contain radial grooves extending from the center hole to the edge of the flow distribution disks. Concentric grooves should be used to connect the radial grooves. By including distribution disks and grooved end plates, the hydraulics of the inlet and outlet will more closely approximate the one-dimensional flow assumption that is used to model contaminant transport in columns.

Side-wall effects

58. Montgomery (1978) investigated the effects of column diameter on settling tests for dredged material and found significant wall effects in columns with diameters less than 20 cm (8 in.). Wall effects in small-diameter columns containing dredged material resulted from bridging of solids that was associated with lateral confinement by the column walls. This bridging phenomenon causes greater densification in the center of the column than along the walls of the column. Comparison of wall effects in small- and large-diameter columns containing dredged material is shown in Figure 6. As shown in Figure 6, there is greater resistance to flow in the center of the column than along the column walls in small-diameter columns due to greater densification toward the center of the column. In large-diameter columns, wall effects are reduced because the distance from the center to the walls is too large for bridging to take place.

59. Since the recommended column diameter is larger than the minimum diameter suggested by Montgomery (1978) and the column diameter-to-particle diameter ratio (assuming an effective particle diameter of 1 mm) is greater than the 200:1 ratio suggested by Sommerton and Wood (1988), wall effects should not be significant. A divided-flow column design involving a double ring at the outlet is therefore not recommended. An investigation of wall effects in the improved column leaching apparatus, however, is recommended to determine the relative effect of column diameter on the hydraulics of the column and to show that wall effects have been adequately minimized.

Recommended Design

60. The recommended column design is shown in Figure 7. The column body is stainless steel tube or pipe. End plates (labeled as top plate and baseplate in Figure 7) are stainless steel plates, 2.54 to 5.08 cm (1 to 2 in.) thick, machined to accept a circular distribution disk and a threaded stainless steel tube fitting. Flow distribution disks are sintered stainless steel, 0.3 cm (1/8 in.) thick with a diameter equal to the inside diameter of the column. The end plates contain eight radial grooves (Figure 8) extending from the hole in the center of the plate to the edge of the flow distribution disk. The grooves are 0.64 cm (1/4 in.) wide and 0.64 cm (1/4 in.) deep. The end plates should have O-rings located to match up with the column body to provide a good seal. The O-rings should be "inert." End plates are attached
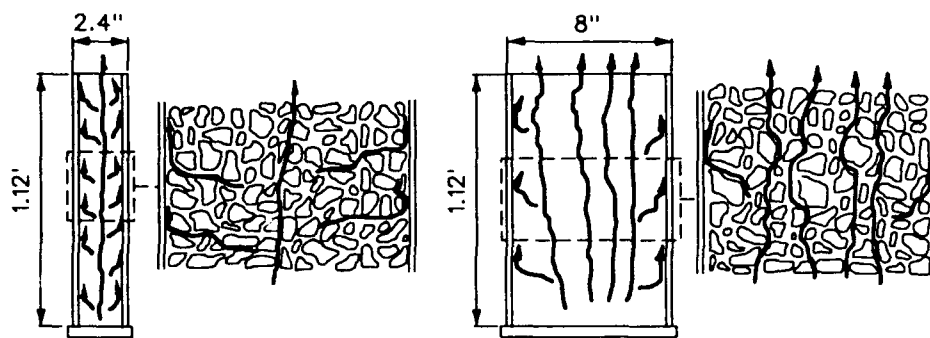
Figure 6. Illustration of wall effects in settling columns (from Montgomery 1978)
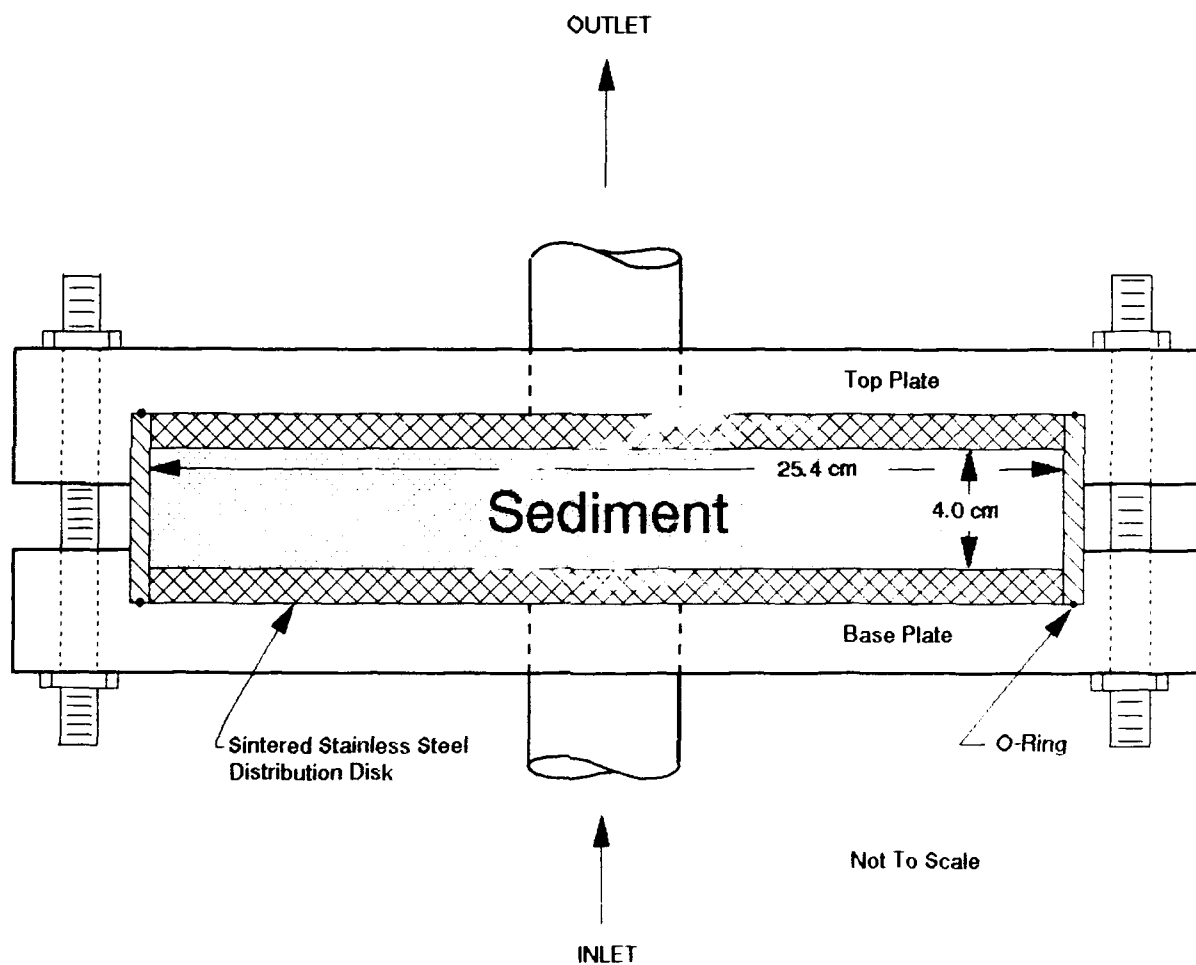
Figure 7. Schematic of improved column leaching apparatus for sediments and dredged material

Concentric Grooves

Radial Groove

INLET/
OUTLET

0.64 cm
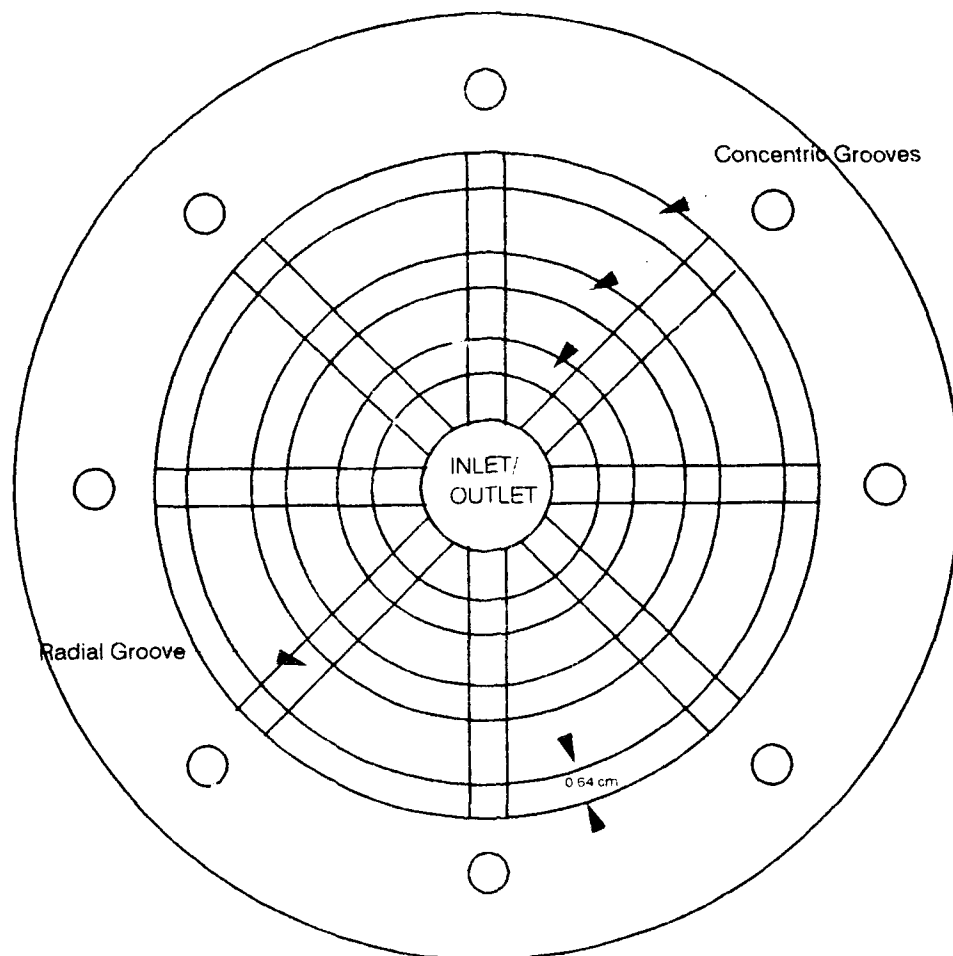
Not To Scale

Figure 8. Schematic of end plate for improved column
leaching apparatus for sediments and dredged material

to the column body by eight 0.6-cm (1/4-in.) threaded rods as shown in
Figure 7. All inlet and outlet tubing should be stainless steel.

61. The thickness of the distribution disk and the diameter of the
inlet/outlet tube can be adjusted to provide one-dimensional flow at the
sediment-distribution disk interface. A theoretical and experimental analysis
of the inlet and outlet hydraulics is needed to show that the design recom-
mended in this report approximates one-dimensional flow at the sediment-
distribution disk interface. It is anticipated that such an analysis would
show that the thickness of the distribution disk needed to provide one-
dimensional flow at the sediment-distribution disk interface is a function of
the diameter of the inlet/outlet tube to the diameter of the distribution disk
ratio.

62. When filling with sediment, it may be necessary to secure the bot-
tom end plate to the column to prevent sediment from becoming trapped between

the O-ring and the column body. To avoid this problem, a temporary anchor consisting of a pipe-riser clamp is recommended. Sediment should be filled to the top of the column body in two or more lifts. Each lift should be vibrated by vibrating the entire column on a vibration table or by some other means so that uniform density is achieved. The final height of sediment should be equal to the height of the column body. Prior to sealing the top end plate, the sediment should be scraped level with a straightedge.

PART IV: SAMPLING AND SAMPLE PRESERVATION

Introduction

63.   As previously discussed, one of the major problems with the column leaching apparatus used at WES is the potential for sample deterioration during leachate collection.   This part of the report describes improved sample collection systems for preserving the chemical integrity of column leachate samples collected over an extended time interval.

64.   The improved sample collection and preservation system is based on plans for the improved column leaching apparatus that specify separate column leaching units for metals and organics.   The collection system is designed to preserve samples accumulated at a slow rate for periods of up to several weeks without adsorption, precipitation, volatilization, or biodegradation losses in the collection vessels.   It is assumed that only the total metals leached from the column are important, and that speciation of the metals for particular or general chemical forms, (i.e., free Cd^{2+} and Cd^{+2} bound to colloidal material) does not have to be maintained in the sample collection system.   Similarly, it is assumed that speciation of the organic chemicals of interest as truly dissolved and sorbed to colloidal matter does not have to be maintained in the sample collection system prior to accessing the collection vessel for analysis of leachate samples.

65.   The collection system is defined as beginning at the column outlet. It is also assumed that leachate leaving the column outlet and entering the collection system represents a solution phase moving through the column such that constituents of interest in the samples can be considered soluble, or, of such a fine colloidal state that they essentially move as if in solution. Under these conditions, it is believed the sample handling and preservation techniques appropriate for aqueous solutions will apply to column leachate.

Sampling and Sample Preservation for Metals

Redox/pH control

66.   The key to preserving metals in water samples is pH control.   The accepted method for preserving water samples for metals analysis is acidification to a pH of 2.0 or less with nitric acid (Ballinger 1979).   Storage specifications state glass or plastic ("....polyethylene with a polypropylene

cap, no liner preferred") containers are acceptable, and a holding time of
6 months is indicated except for mercury where the holding time is given as
38 days in glass and 13 days in hard plastic. Refrigeration and storage of
the sample under an inert atmosphere are not required for metal preservation
purposes.

67. Monitoring or controlling redox potential of the collected leachate
is not required since redox potential does not affect metal solubility at pH
less than 2.0. Thus, for purposes of sample preservation for metals analyses,
only pH and container composition are important.

68. Depending on the column outlet design, an inert atmosphere for the
sample collection assembly may be needed. If the outlet tube is not submerged
in collected leachate, oxidizing conditions surrounding the outlet tube could
cause problems. As leachate slowly emerges from the outlet and droplets form,
the solution is leaving an anaerobic environment. If the droplets linger on
the outlet in the presence of air prior to dropping to an acidified solution
below, some of the anticipated high amounts of ferrous iron may oxidize and
precipitate on the outlet because of the relatively rapid rate at which fer-
rous iron can oxidize in air and the anticipated slow drop formation on the
column outlets. Iron precipitation may possibly occlude other metals moving
past this point as well, removing metals that would otherwise be in the pre-
served leachate sample.

69. For a collection system in which the outlet from the column is
submerged in collected leachate, an inert atmosphere for the collection system
is not necessary, and the collection system could be open to the atmosphere.
The system should be designed to prevent evaporative losses of leachate over
extended collection periods.

70. Because the volume of acid added for preservation of metals will
dilute the metals concentrations in the collected leachate, the volume added
should be small relative to the volume of leachate preserved. The amount of
acid required depends on column flow, acid normality, initial pH of the leach-
ate, the buffering capacity of the leachate (alkalinity), and the target pH
for preserved sample. Assuming an initial pH of 7, a target pH of 1.8, an
alkalinity of 1,000 mg/ℓ (as $CaCO_3$) (Mang et al. 1978), a flow of 1,115 ml per
2 weeks, about 18 ml of 1.0 normal nitric acid will be required to maintain pH
for metal preservation over a 2-week interval. Metal concentrations will be
diluted by less than 1.6 percent. This level of dilution is within the

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precision of chemical analytical techniques for metals and is therefore acceptable.

Recommended design

71. The recommended design is an adaptation of the apparatus used to study the chemistry of nutrients and contaminants in soil and sediment suspensions under controlled redox potential and pH conditions (Gambrell et al. 1984). However, the redox potential measurement and control components of the apparatus used by Gambrell et al. (1984) are not needed for preservation of metals in column leachate. Any metals in an acidified solution as described above will be adequately preserved.

72. An all-glass vessel with an auxiliary access port, pH control (automatic or manual), continuous stirring, and submergence of the column outlet initially in pH-1.8 water is recommended. A schematic diagram of the recommended sample collection and preservation system for metals is shown in Figure 9. Each of the design features is discussed below.

73. Submerged outlet. The column outlet should be fitted with small-diameter glass or plastic tubing so that the lower end is submerged at all times. A small amount of distilled-deionized water at pH 1.8 will have to be placed in the collection vessel initially to keep the tip of the tubing submerged.

74. All-glass vessel. An all-glass vessel offers longer acceptable holding times for mercury, and may be less permeable to oxygen than some plastic containers, though oxygen permeability should not be a problem.

75. pH control. Since the key to preserving metals in the collected leachate is pH control, automatic adjustment of pH should be considered. Automatic adjustment of pH will reduce labor costs and should offer more uniform control than is possible with manual control. Automatic pH control is accomplished by having a permanently installed, combination pH electrode in the collected leachate coupled to a commercially available pH/redox controller. Whether pH or redox potential is controlled is just a matter of the electrodes used and setting the meter panel switch to measure pH or electrical potential in millivolts. As the pH increases above some preset value (i.e., 1.8), the controller activates a low-flow-rate peristaltic pump that slowly meters a dilute solution of nitric acid into the collection assembly. When the pH of the solution reaches the desired level again, the pump automatically shuts off. A controller with an expanded-scale pH mode and a very low-flow rate pump should give the best performance.
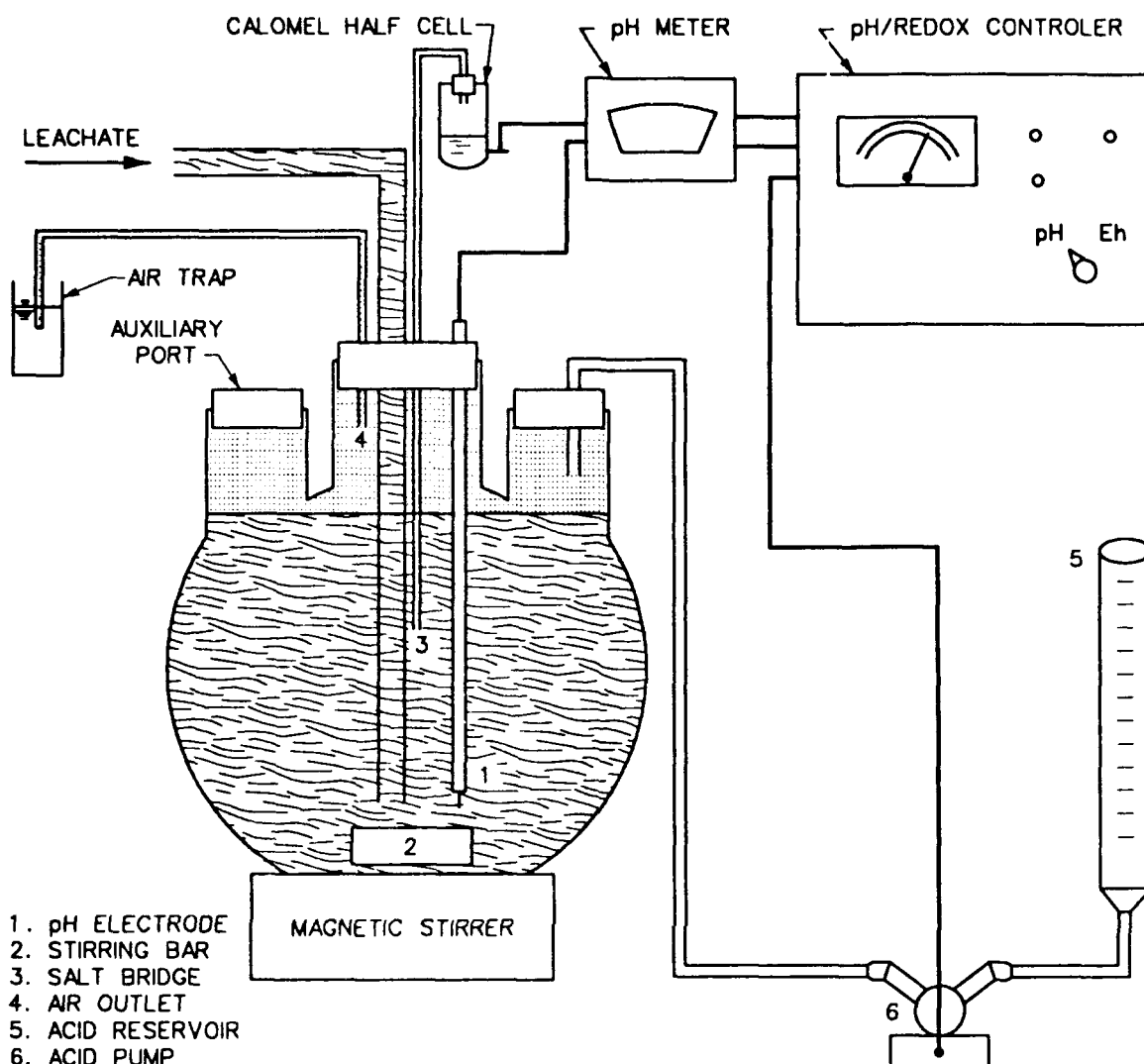
Figure 9. Apparatus for collecting and preserving column
leachate for metals analysis

76. A wide variety of pH/redox controllers are commercially available.
Models are available with built-in recorders to continuously record pH (or
redox potential).

77. Calibration of the pH electrode and meter should probably be
checked at 2- to 3-day intervals initially. Then, if drift does not seem to
be a major problem, weekly recalibrations may be sufficient. Some electrode
designs are more susceptible to gradual clogging of the reference solution
junction by the sample. When junction potential changes, calibration of the
electrode is affected. A small amount of water will have to be placed in the
collection vessel initially to keep the electrode submerged.

78. As previously discussed, the dilution associated with addition of acid for sample preservation may be minimal. Even if the dilution proves to be minimal, the amount of acid used to preserve samples should be measured and recorded. The amount of acid added can be measured in either of two ways. The first is to connect a clock to the same electrical outlet from the pH controller that powers the acid delivery pump. Knowing the pump flow rate and the accumulated pump operating time, the volume of acid added to the leachate collected can be determined. Sophisticated clocks are available, but an inexpensive analog clock will suffice. An alternative and simpler method is to record volume changes in a calibrated acid reservoir (with precautions to prevent evaporation) for each collection assembly. A simple buret would serve this purpose.

79. It is suggested that the acid reservoir be placed below the collection reservoir. If problems develop, there will be no possibility of uncontrolled drainage of acid from the reservoir into the collection vessel. Insufficient acid delivery for a few hours or overnight due to pump failure or other problems is not as critical as accidental delivery of too much acid.

80. Although continuous, automatic measurement and adjustment of pH should be considered, manual pH monitoring and adjustment is feasible and offers some advantages over automatic pH monitoring and adjustment. At the low flow rates anticipated from the columns, pH could be read at twice-a-day intervals and small amounts of acid added manually to maintain the pH over a narrow range of 1.6 to 1.9. Manual monitoring and adjustment will minimize the clogging problem previously discussed for automatic monitoring and adjustment. In addition, with manual monitoring and adjustment there is no potential for flooding of the collection vessel with acid.

81. <u>Continuous stirring.</u> Continuous stirring offers the advantage of maintaining a uniformly acidified condition. Stirring is also necessary to insure that the pH measured at a pH sensor is representative of the entire leachate volume. Stirring should be accomplished with a magnetically coupled stirring device. A Teflon-coated magnet in the collection vessel where the collection vessel is semi-permanently placed on a motor-driven magnetic stirrer is recommended. Some motor-driven magnetic stirrers get warm on top and transfer heat into the solution being stirred. This may not be a significant problem, but unnecessary heating of the sample should be avoided. Units with small cooling fans in the motor housing are available that transfer the motor heat out the back of the unit. Also, in units without cooling fans, some have

been noted to run cooler than others. The differences in heat generation should be considered in selection of equipment.

82. Auxiliary access port. The collection assembly should be designed with an auxiliary port that can be sealed. There are several activities that an auxiliary access port will facilitate. One is the collection of subsamples of the leachate during the course of the run if necessary. Another use might be for calibration of the pH electrode, if automatic pH monitoring and adjustment is used. It may be useful to re-calibrate a permanently installed electrode by temporarily inserting a calibrated electrode into the system through an auxiliary port.

Operating suggestions and precautions

83. One potential problem is that soluble or colloidal humic matter in the leachate may precipitate, coagulate, or polymerize into particulate matter that settles upon lowering pH for preservation of metals. Precipitation, coagulation, and/or polymerization of colloidal humic matter can occur in stored samples over a period of time, and especially in aqueous sediment extracts. In stored samples, this material is sometimes observed as a slight discoloration of sample container walls or as a floc in the bottom of the sample container. It is not known if metals are bound to the humics by charge interactions, occluded in a particulate phase, or displaced from all the charged sites on humic materials by the high concentration of protons in solution. Unless work is done to demonstrate otherwise, the possibility of some fraction of the metals becoming associated with humic matter should be considered.

84. Where continuous stirring is used, loss of metals associated with precipitated, coagulated, and/or polymerized colloidal humic material may or may not be a problem. To minimize potential problems, the collection assembly should be shaken or stirred well to insure that particulates are suspended prior to transferring sample from the collection assembly to bottles for analysis. If visible particulates are present or form in the storage bottles, brief sonification (ultrasound) with a stainless steel probe directly in the sample bottles will break up the humic particulates to a colloidal state making the sample suitable for analysis by inductively coupled plasma (ICP) or flame atomic absorption (AA). Analysis of acidified pure water so treated has indicated no detectable metals contamination within the detection limits of the ICP by direct insertion of the stainless steel probe into the solution. If there is concern about this for more sensitive analytical techniques (i.e.,

graphite furnace AA), placing a sample bottle directly in an ultrasonic bath for a longer period of time should break up the humic particulates to a colloidal state.

Sampling and Sample Preservation for Organics

85. The organics of interest include but are not limited to polynuclear aromatic hydrocarbons, chlorinated hydrocarbon pesticides, and chlorinated industrial organics, in particular PCBs. Because of the anticipated low concentrations of these organics in leachate from CDFs and laboratory columns used to simulate CDFs, relatively large quantities of water on the order of liters must be collected and extracted to have sufficient levels of organics to analyze. The organic components of interest must be preserved so that the chemical composition of leachate samples remains unchanged. The recommended sample collection and preservation method is an adaptation of the condenser and solvent reservoir of a Nielson-Kryger steam distillation apparatus.

Background on steam distillation

86. Figure 10 shows a typical steam distillation unit for extracting sediments. Steam distillation has been successfully used to extract PCBs and chlorinated hydrocarbon pesticides from sediments and water samples. In steam distillation of sediments, water is added to a wet sediment in a boiling flask connected to a specially designed condensing unit. A heating mantle is used to bring the sediment-water mixture to a rolling boil, usually for a period of many hours. The non-polar compounds of interest co-distill with water, and both water and non-polar organics condense on the cool walls of the condenser. The high rate of condensation results in water and non-polar organics being "rinsed" by gravity flow through a region containing a relatively small volume (i.e., 10 to 15 ml) of a non-polar solvent such as toluene or hexane. Water, having greater density than the solvent and being sparingly soluble in the solvent, moves downward through the stationary solvent layer and is returned to the boiling flask through an overflow tube that permits only water to return. The non-polar compounds of interest are stripped from the water as the water moves through the solvent and are retained in the solvent. Upon completion of the distillation, a valve on a side arm tube connected to the solvent region is used to recover the solvent containing the components of interest.

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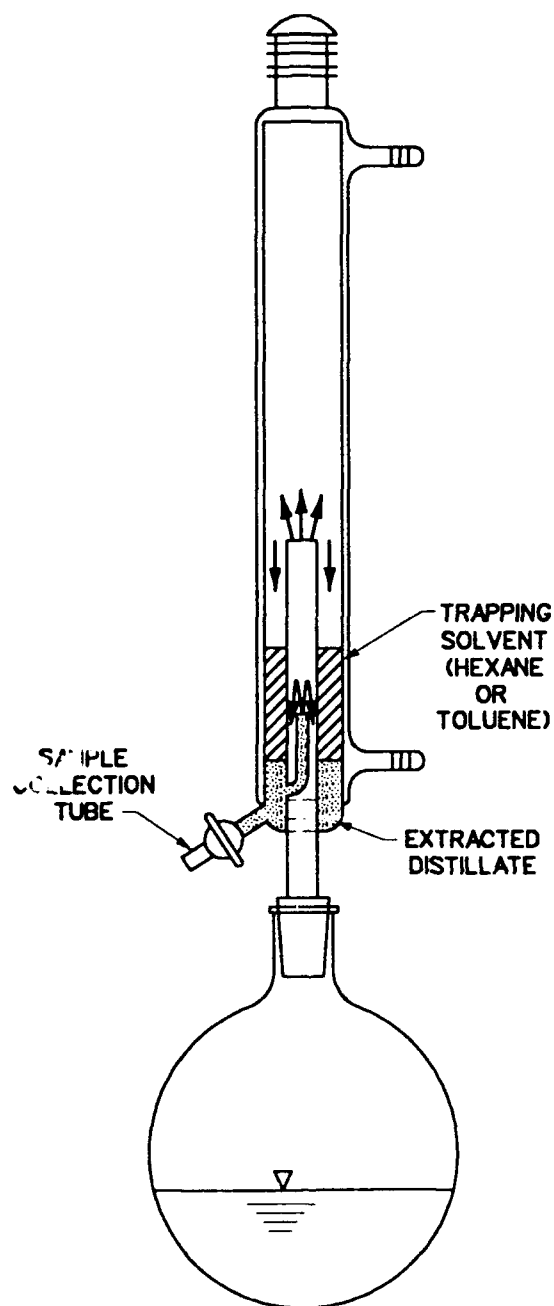
Figure 10. Nielson-Kryger steam
distillation apparatus

87. Swackhamer and Armstrong (1986) used steam distillation to extract
PCBs in sediments of Lake Michigan and several lakes in Wisconsin. Wet sedi-
ment (100 to 200 grams) was extracted into hexane, and the hexane extracts
were cleaned by elution through columns containing alumina, silica gel, and
copper. The reported recoveries were greater than 90 percent for Aroclors
1242, 1248, and 1254, and about 60 percent for Aroclor 1260.

88. Veith and Kiwus (1977) showed that steam distillation recovered from 97.9 to 100 percent of Aroclors 1016, 1242, 1248, and 1254 from spiked water samples with boiling times of 45 to 60 min. The recovery percentages with 60 min extraction for pp'DDE, heptachlor epoxide, and mirex were 104.0, 89.9, and 89.5 percent, respectively.

89. Bellar, Lichtenberg, and Lonneman (1980) studied three sediment extraction procedures for PCBs and pesticides: soxhlet extraction, sonification, and steam distillation. Hexane (15 ml) was used as the trapping solvent and distillation (boiling of sediment-water slurry) was conducted for various periods of time ranging from 3 to 71 hr. The methods gave equivalent recoveries for PCB-spiked samples (109 percent for soxhlet, 103 percent for two-step sonification, and 99 percent for 4-hr steam distillation). From contaminated environmental samples, soxhlet extraction gave the best recovery of PCBs. For soxhlet extraction arbitrarily ranked at 100-percent recovery, 71-hr steam distillation gave 75-percent recovery. Lower recoveries by steam distillation relative to soxhlet extraction were probably due to incomplete removal of contaminant from the sediment solid phase rather than inefficient contaminant trapping as condensed water moved through the solvent layer.

90. Incomplete removal from sediment solids is not a factor in the analysis of leachate samples since no sediment solids in the usual sense are present. Steam distillation of straight aqueous leachate samples should give excellent recoveries with short boiling times. Advantages of the method include efficient recovery of Aroclors and similar compounds, concentration of the components of interest in a small volume of solvent, and, minimization of extract cleanup requirements as humic materials do not co-distill with the steam (Eisenreich, Hollod, Johnson, and Evans 1980).

Recommended apparatus

91. A Nielson-Kryger column (Figure 10) with a boiling flask of suffi-cient capacity to hold the leachate volume required for analysis is recom-mended for collection and preservation of column leachate for organic analysis. The trapping solvent can be hexane or toluene. A series of small boiling flasks instead of one large boiling flask could be used by switching to an empty flask as one becomes full.

92. The leaching column drain tube should be positioned so that the leachate directly enters the solvent layer in the Nielson-Kryger column. This arrangement will minimize ferrous iron oxidization and precipitation at the end of the tube and the subsequent possibility that the precipitate formed

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would occlude organics of interest (Pionke and Chesters 1973). The lower unit of the collection system (boiling flask) should be kept in a low-light environment and possibly refrigerated if there is concern about degradation over extended collection times.

93. The Nielson-Kryger column should be "operated" cold during the collection period. At the end of a run, the entire collection unit (condenser and boiling flask) is connected to a heating mantle and chilled water lines. The unit would then be operated to steam distill and extract the organics of interest.

Operating suggestions and precautions

94. Since distillation will not be initiated until the end of leachate collection, the system also has the potential for separate determination of "dissolved" non-polar organics and those bound to colloidal particulates. In natural surface and interstitial waters, it has been shown that non-polar chlorinated hydrocarbons are not present primarily as free molecules dissolved in water, but as complexes with soluble humic materials or insoluble colloidal particulates that move as if dissolved (Wershaw, Burcar, and Goldberg 1969; Tulp and Hutzinger 1978; Myers and Brannon 1988a). Analysis of the trapping solvent before and after boiling may reveal something about free versus bound non-polars in the leachate. The dissolved non-polars may be efficiently extracted in the solvent layer as the leachate slowly passes through on its way to the boiling flask. Non-polars bound to colloidal humic particulates may pass through the solvent layer with the water to the boiling flask.

95. It is not known how efficiently these complexes are trapped during passage through a hexane or toluene layer. Some proportion of the colloidal humic particulates moving with water may be retained in the solvent layer, or, some of the non-polar organics of interest may desorb in the solvent layer. Whether the fractionation of organic contaminants between dissolved and particulate-bound forms could be measured this way would have to be experimentally verified.

96. Less methods development work has been done with aqueous anaerobic sediment extracts than with surface water sample extracts. Column leachate samples may not quantitatively respond to the extraction methods the same as typical surface water samples. Since non-polar organics may be more tightly bound to colloidal matter in anaerobic sediment leachate than in surface water samples, the recommended collection and extraction methods for aqueous

anaerobic sediment leachates needs to be verified. Relatively simple and quick studies using ^{14}C-labeled compounds could be used to verify methods.

PART V· SUMMARY AND RECOMMENDATIONS

97. This report addressed three specific areas for design of an improved column leaching apparatus for sediments and dredged material. These areas included a review of the literature, an improved column design, and improved sampling and sample preservation procedures.

Literature Review

98. Three areas of research involving column studies are reviewed in this report: soil columns, solid waste columns, and sediment and dredged material columns. The review focused on column design and operating parameters such as flow rate.

99. The literature review showed a wide variety of column designs, but nearly all involve percolating water through a vertical column. Studies with sediments and dredged material showed that the performance of column designs based on the type of column used in most soil and solid waste studies is not satisfactory, primarily due to insufficient flow.

Recommendations

100. A number of recommendations are made specifically addressing improved column design and sampling and sample preservation procedures. These recommendations may be summarized as follows:

Column design

    a. Column diameter of 25.4 cm and column length of 4 cm are recommended. Specific design features are shown in Figures 7 and 8.

    b. Flow should be controlled using a constant volume pump.

    c. An upflow mode should be used with distribution disks at both the top and bottom of the sediment column.

    d. Pore water velocity in column leach tests for sediments and dredged material should be less than 1 E-05 cm/sec.

Sampling and sample preservation

    a. For metals, an adaptation of the apparatus used to study the chemistry of soil and sediment suspensions under controlled redox potential and pH conditions is recommended (Figure 9).

    b. For organics, the recommended collection and preservation method involves use of a trapping solvent (hexane or toluene) in an adaptation of a Neilson-Kryger steam distillation

apparatus with a boiling flask of sufficient capacity to hold the leachate volume required for chemical analysis (Figure 10).

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Table 1

Characteristics of Selected Soil Column*

Reference	ID, cm	L, cm	V, cm/sec	Mode	Material	Medium	Solute
Davidson and Chang 1972	7.6	30.0	--	--	Glass	Norge loam	Picloram
Hornsby & Davidson 1973	7.6	30.0	$(1.6\text{-}15)E\text{-}4$	Up	Lucite	Clay loam	Fluometuron
Cassel et al. 1974	30.0	80.0	$1\ E\text{-}5$	Down	Steel	Clay loam	Nitrates
Van Genuchten, Davidson, and Wierenga 1974	7.6	30.0	$1.6E\text{-}4\text{-}1.7E\text{-}3$	Down	--	Norge loam	Picloram
Wierenga et al. 1975	75.0	150.0	--	Down	Techite	Clay loam	Cations/ Anions
O'Connor, Van Genuchten, and Wierenga 1976	5.4	30.0	$(1.1\text{-}4.1)E\text{-}4$	Down	Plexiglas	Clay loam	2,4,5-T
Ardakani & McLaren 1977	10.2	44.0	$(0.06\text{-}21)E\text{-}3$	Down	PVC	Sandy loam	Ammonium
Wagenet, Biggar, and Nielson 1977	7.6	15-30	$8.6\ E\text{-}5$	Down	Plastic	Silty clay loam	Nitrogen
Hoffman & Rolston 1980	7.5	2.4-5.1	$(0.64\text{-}3.4)E\text{-}4$	Down	Plastic	Soils	Phosphorus
O'Connor et al. 1980	75.0	150.0	$1.2\ E\text{-}5$	Down	--	Clay loam	2,4,5-T
Rao et al. 1980	7.6	30.0	$(0.52\text{-}31)E\text{-}3$	Ref-3	Plexiglas	Fine sand	Tracers
Akratanakul, Boersma, and Klock 1983	5.7	6.0	$(4.3\text{-}9.0)E\text{-}4$	Down	Plastic	Silty sand	Cadmium
Nkedi-Kizza et al. 1983	7.6	5.0	$(0.38\text{-}16)E\text{-}4$	--	Plastic	Clay	Tracers
De Smedt and Wierenga 1984	5.4	30.0	$1.5\ E\text{-}3$	Down	Plexiglas	Glass beads	Calcium
Goerlitz 1984	1.5	26.6	$2.6\ E\text{-}3$	--	SS	Glacial till	Organics
Hutzler, Crittenden, and Gierke 1986	5.1	30.5	$(3.3\text{-}10)E\text{-}3$	Up	Glass	Sandy loam	TCE & Bromoform

* Note: ID = inside diameter; L = length; V = average pore water velocity; SS = stainless steel; PVC = polyvinylchloride; MODE = direction of flow; TCE = trichloroethane.

Table 2

Characteristics of Selected Solid Waste Columns*

Reference	ID, cm	L, cm	WAR, cm/d	Mode	Material	Waste
Mahloch, Averett, and Bartos 1976	10.2	121.00	0.86	Down	Plastic	HW
Steng 1976	183.0	366.00	0.11	Down	Steel	MSW/IW
Fungaroli & Steiner 1979	183.0†	396.00	0.02	Down	Steel	MSW
Myers et al. 1979	183.0	366.00	0.18	Down	Steel	MSW/HW
Walsh & Kinman 1979	183.0	366.00	0.056-0.22	Down	Steel	MSW/HW
Dodd et al. 1981	11.0	17.50	0.26-1.22	Down	Polycarbonate	Fly ash
Jackson, Benedik, and Jackson 1981	12.7	7.60	--	Down	Glass	Fly ash
Darcel 1983	3.5	--	31.00	Down	Plexiglas	HW
Maskarinec & Brown 1983	2.5	45.00	--	Up	Glass	MSW/HW/ Sand
Stevens, Jenkins, and Wilson 1983	15.0	150.00	0.25	Down	Glass	MSW
Jackson, Garrett, and Bishop 1984	5.0	60.0	68.00	--	Glass	HW/Sand
Young et al. 1984	10.0	6.88	367-550	Down	Glass	Fly ash
Francis et al. 1986	180.0	360.00	1.00	Down	Concrete	MSW
Pohland 1987	80.0	300.00	0.13	Down	Steel	MSW/HW

* Note: ID = inside diameter; L = length; MSW = municipal waste; WAR = water application rate; HW = hazardous waste; IW = industrial waste; † = square surface area.

Table 3

Design Calculations for Elution of 1 ℓ per 2 Weeks*

$n = 0.7$ $Q = 13$ ℓ/6 months
$P_v = 10$ $t = 6$ months

Length cm	Diameter cm	v E-05 cm/sec
12	14.0	0.763
11	14.7	0.700
10	15.4	0.636
9	16.2	0.572
8	17.2	0.509
7	18.4	0.445
6	19.9	0.382
5	21.7	0.318
4	24.3	0.254

* Note: n = porosity; Q = flow; P_v = number of pore volumes eluted; t = time period for elution; v = average pore water velocity.

Table 4

Design Calculations for Elution of 1 ℓ per Week*

n = 0.7   Q = 26 ℓ/6 months

P_v = 10   t = 6 months

Length cm	Diameter cm	v E-05 cm/sec
12	19.9	0.763
11	20.7	0.700
10	21.7	0.636
9	22.9	0.572
8	24.3	0.509
7	26.0	0.445
6	28.1	0.382
5	30.8	0.318
4	34.4	0.254

* Note: n = porosity; Q = flow; P_v = number of pore volumes eluted.